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October 29, 2020

Ms. Karlene Fine
Executive Director
North Dakota Industrial Commission
State Capitol, 14th Floor
600 East Boulevard Avenue, Department 405
Bismarck, ND 58505-0840

Dear Ms. Fine:

Subject: Quarterly Project Status Report Entitled "Low-Pressure Electrolytic Ammonia Production"; Contract No. R-036-45; EERC Fund 23228

Attached is a copy of the subject project status report for the period of July 1 through September 30, 2020.

If you have any questions, please contact me by phone at (701) 777-2982 or by e-mail at taulich@undeerc.org.

Sincerely,

DocuSigned by:
Ted Aulich
89B1B8E4D0E7430...

Ted R. Aulich
Principal Process Chemist
Fuels and Chemicals

TRA/kal

Attachment

c/att: Andrea Holl Pfennig, North Dakota Industrial Commission

LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION

Quarterly Project Status Report

(for the period of July 1, 2020, through September 30, 2020)

Prepared for:

Karlene Fine

North Dakota Industrial Commission
State Capitol, 14th Floor
600 East Boulevard Avenue, Department 405
Bismarck, ND 58505-0840

Contract No. R-036-45

Prepared by:

Ted R. Aulich

Energy & Environmental Research Center
University of North Dakota
15 North 23rd Street, Stop 9018
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October 2020

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION
Quarterly Project Status Report
July 1 – September 30, 2020

EXECUTIVE SUMMARY

This quarterly report summarizes July–September 2020 progress made toward achieving milestones and objectives of the low-pressure electrolytic ammonia (LPEA) project under way at the University of North Dakota Energy & Environmental Research Center (EERC). Partners on the 3-year (June 2018 – July 2021) project include North Dakota State University (NDSU), Nel Hydrogen (formerly Proton OnSite), and North Dakota Industrial Commission. The project goal is to demonstrate an ammonia production energy reduction of at least 16% by replacing state-of-the-art (2018) high-pressure Haber–Bosch-based ammonia synthesis with the EERC-developed LPEA process. Achieving this energy reduction goal requires improving the proton conductivity, gas impermeability, and durability of the EERC–NDSU-developed polymer–inorganic composite (PIC) proton exchange membrane, a critical LPEA process component capable of high-rate proton transfer at 300°C. Key accomplishments of the July–September 2020 quarter include the following:

- A modified version of "IPC2,"—the inorganic proton conductor material utilized as the basis for the PIC membrane—was demonstrated to provide significantly higher proton conductivity than the original IPC2. The modified version, referred to as IPC2-A2, was prepared by altering the surface chemistry of IPC2 particles, resulting in 57% higher proton conductivity at 300°C.

- A specialized airbrushing technique was developed and optimized for application of electrode (anode and cathode) layers to a film-cast 75-µm-thick PIC membrane comprising 75 weight% IPC2 and 25% polybenzimidazole. The technique was utilized to fabricate an identical series of membrane–electrode assemblies for use in evaluating LPEA unit-cell performance based on ammonia synthesis rate and current efficiency.

The EERC holds an unwavering commitment to the health and well-being of its employees, partners and clients, and our global community. As such, precautionary measures have been implemented in response to COVID-19. Staff continue to carry out project-related activities remotely, and personnel supporting essential on-site laboratory and testing activities are proceeding under firm safety guidelines. Travel has been minimized, and protective measures are being undertaken for those who are required to travel. At this time, work conducted by EERC employees is anticipated to progress with minimal disruption. Challenges posed by economic variability will be met with open discussion between the EERC, the U.S. Department of Energy Project Manager, and other partners to identify solutions. The EERC is monitoring developments across the nation and abroad to minimize risks, achieve project goals, and ensure the success of our partners and clients.

LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION
Quarterly Project Status Report
July 1 – September 30, 2020

PROJECT GOALS/OBJECTIVES

The project goal is to demonstrate an ammonia production energy reduction of 16% by replacing state-of-the-art (2018) high-pressure Haber–Bosch (HB)-based ammonia synthesis with the Energy & Environmental Research Center (EERC)-developed low-pressure electrolytic ammonia (LPEA) process, as shown in Figure 1. To achieve the 16% production energy reduction target will require improving the LPEA process, which will require improving the polymer–inorganic composite (PIC) proton exchange membrane (PEM) on which the LPEA electrochemical cell is based. As a result, the proposed project is focused on improving the performance and durability of the PIC membrane, with the objective of producing a membrane that exhibits the following properties:

- Proton conductivity of $\geq 10^{-2}$ Siemens/centimeter (S/cm) and gas permeability of <2% at a minimum temperature of 300°C.
- Ability to sustain $^{-2}$ S/cm proton conductivity for at least 1000 hours (h).

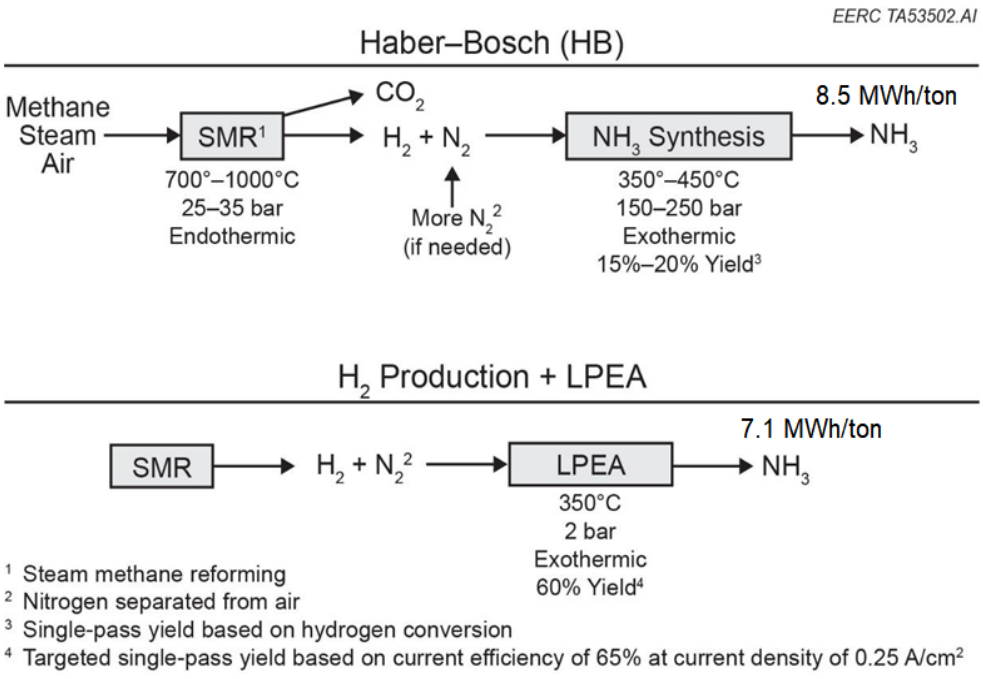

Figure 1. State-of-the-art (2018) HB versus LPEA-based NH_3 production.

- Mechanical strength (at 300°C) comparable to that of a commercial proton exchange-based electrolyzer membrane.

- As measured in a membrane–electrode assembly (MEA) at a minimum temperature of 300°C, current efficiency of ≥65% for NH_3 formation at a current density of ≥0.25 amps/cm^2 (A/cm^2), NH_3 production energy efficiency of ≥65%, and ≤0.3% performance degradation per 1000 h of operation.

BACKGROUND

In support of U.S. Department of Energy (DOE) Energy Efficiency and Renewable Energy (EERE) Advanced Manufacturing Office (AMO) goals to reduce life cycle energy consumption of manufactured goods and more cost-effectively use hydrogen in manufacturing processes, this project is focused on optimizing and demonstrating the improved efficiency (versus HB ammonia production) of the EERC-developed LPEA production process. Because it does not require the high pressure and high recycle rate (because of low single-pass ammonia yield) of the HB process, LPEA offers the potential for significant reduction in both energy consumption and cost. Partners on the proposed project are North Dakota State University (NDSU), Nel Hydrogen (Nel) (formerly Proton OnSite), the University of North Dakota Chemistry Department (UND Chemistry), and the North Dakota Industrial Commission (NDIC). The LPEA process is based on an innovative EERC-developed PIC high-temperature PEM. The process operates at ambient pressure and a temperature of 300°C and uses inputs of hydrogen, nitrogen, and electricity to make ammonia. The EERC demonstrated LPEA process viability in ammonia formation tests conducted using a 0.2-watt electrochemical cell built around an early-stage PIC membrane.

To meet the above-listed membrane performance and durability specifications, the project initially targeted fabrication—via a “co-electrospinning” technique—of a PIC membrane comprising “core–shell” inorganic proton conductor–polybenzimidazole (IPC–PBI) proton-conducting nanofibers contained within and aligned perpendicularly to the plane of a PBI matrix/membrane, as shown in Figure 2. Because each fiber core would comprise a chain of IPC particles in contiguous contact with one another throughout the chain length, each fiber would essentially function as a high-efficiency proton transport conduit running straight through the membrane. However, during Budget Period 1 (BP1) of the project, an alternative IPC was identified that offered significantly improved proton conductivity, stability, and durability—at 300°C—than the originally proposed IPC. Because this new IPC (IPC2) encompasses chemical and physical properties not readily amenable to co-electrospinning with PBI to yield core–shell nanofibers, new methods for IPC2 deployment in PBI matrix are being pursued. Primary focus is on film-casting (also referred to as solution-casting) a colloidal suspension of optimally sized IPC2 particles in a solution comprising PBI dissolved in dimethylacetamide (DMAc).

Following fabrication of a PIC membrane that meets performance and durability specifications, the membrane—along with selected anode and cathode catalysts—will be used to construct experimental MEAs. MEAs will be incorporated into LPEA unit cells that will be evaluated based on NH_3 formation efficiency and durability, with the objective of identifying an

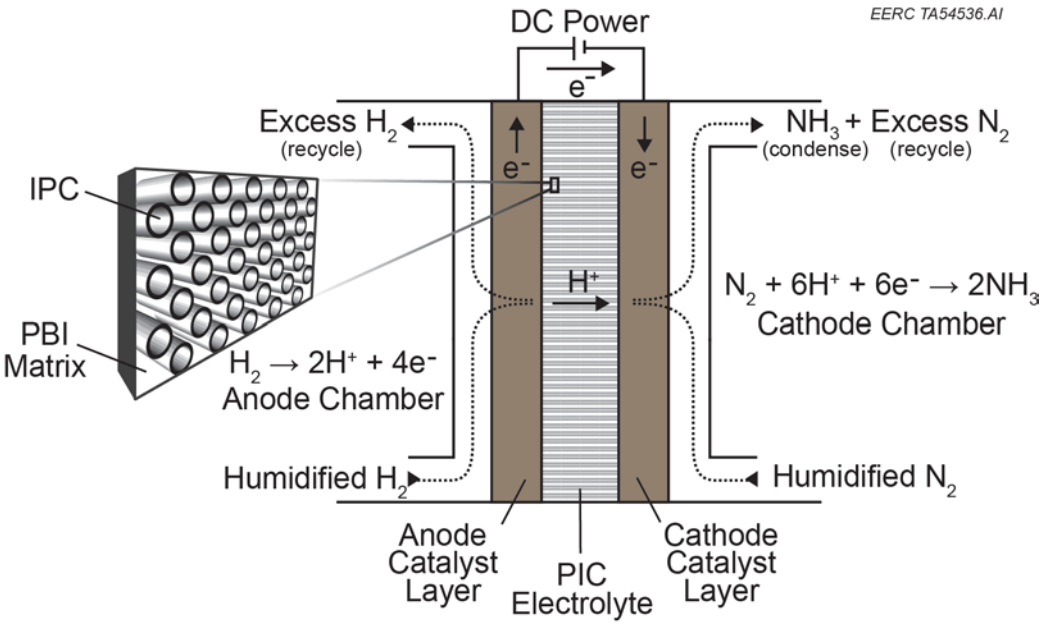

Figure 2. LPEA process.

optimal MEA configuration. The optimal MEA configuration will be used as the basis for building a stack of several LPEA unit cells that will compose an LPEA system capable of producing at least 100 grams/day (g/d) of NH_3. The 100-g/d LPEA system will undergo optimization and then be used to demonstrate NH_3 synthesis (from H_2) at the LPEA target production energy input requirement of 0.8 megawatt hours (MWh/ton), which would translate to a total (H_2 production plus NH_3 synthesis) LPEA-based NH_3 production energy input requirement of 7.1 MWh/ton, the project-targeted goal. LPEA system operation and performance data will be used to perform a techno-economic evaluation of the LPEA-based NH_3 production process.

ACCOMPLISHMENTS

Key accomplishments during the July–September 2020 reporting period include the following:

- A modified version of "IPC2,"—the inorganic proton conductor material utilized as the basis for the PIC membrane—was demonstrated to provide significantly higher proton conductivity than the original IPC2. The modified version, referred to as IPC2-A2, was prepared by altering the surface chemistry of IPC2 particles, resulting in 57% higher proton conductivity at 300°C.

- A specialized airbrushing technique was developed and optimized for application of electrode (anode and cathode) layers to a film-cast 75-µm-thick PIC membrane comprising 75 weight% IPC2 and 25% polybenzimidazole (PBI). The technique was

utilized to fabricate an identical series of MEAs for use in evaluating LPEA unit-cell performance based on ammonia synthesis rate and current efficiency.

PROGRESS AND STATUS

Task 1 – Project Management

Table 1 summarizes project task status. As shown, all remaining tasks are behind schedule. These progress delays resulted from 1) a tentative BP2 start while awaiting official approval of BP2 funding and 2) a directive issued March 15 by UND President Dr. Joshua Wynne (in response to COVID-19 spread concerns) instructing all nonessential EERC employees to work remotely until further notice, which restricted project laboratory activities. Similar restrictions were implemented at roughly the same time by project partners NDSU and Nel. Labs at all project locations have since been reopened (with social distancing-based staffing capacity limits), but EERC lab work was shut down for 2 weeks during this reporting period due to COVID-19 infection of an LPEA project researcher. The researcher recovered, no colleagues were infected, and EERC lab work has resumed. In addition to the above-mentioned schedule challenges, an LPEA project scientist (employed as a postdoc) abruptly resigned 1 year prior to his planned appointment end date to take a position at the University of Hawaii. The EERC is working to fill the resulting technical project staff vacancy.

Table 1. Task Schedule – BP2

Task No.	Task Description	Task Completion Date			Task Progress Notes
		Original Planned	Revised Planned	% Complete	
1	Project Management	14 June 2021		58	
3	Optimize IPC and PIC membrane performance and durability	14 Dec. 2020		65	Behind schedule
5	Screen cathode catalysts, fabricate MEAs, deploy MEAs in unit cell for LPEA process optimization	14 Dec. 2020		40	Catalysts screened; MEA and unit cell work behind schedule
6	Design, fabricate, and optimize 100-g/d LPEA system; acquire data for techno-economic analysis	14 March 2021			Not started
7	Conduct techno-economic analysis	14 June 2021			Not started

Because the project is significantly behind schedule with less than 8 months remaining until the 14 June 2021 end date, the EERC plans to formally request a no-cost project extension of 12 months, which—if approved—would reset the project end date to 14 June 2022. Although it is anticipated today that project milestone and deliverable requirements will be met and the project successfully completed within an additional 6–8 months, a 12-month extension will allow accommodation of any unforeseen schedule disruptions.

Task 3 – Optimize IPC and PIC Membrane Performance and Durability

After synthesis, high-temperature (800°C) annealing, and grinding via mortar and pestle, IPC2 particles range in size from about 200 nm to 20 μm and exhibit irregular and often highly angular (as opposed to spherical) morphologies. To make a PIC membrane, IPC2 particles are suspended in a solution of PBI in DMAc and film cast to yield an approximate 70-μm-thick membrane with an IPC2/PBI mass ratio of 75/25. Because of the wide size distribution and nonuniform shape of IPC2 particles, the resulting PIC membrane comprises highly diverse (in size and shape) microstructures, which translates to nonoptimum membrane proton conductivity, structural integrity, and gas impermeability (Columban, 1992). Methods being investigated for producing smaller (≤ 200-nm) more spherical IPC2 particles are 1) high-power probe sonication and 2) planetary ball milling in fluid (water, ethanol, or other). Figure 3 shows scanning electron microscope (SEM) photos of IPC2 particles before and after probe sonication for 20 minutes at a

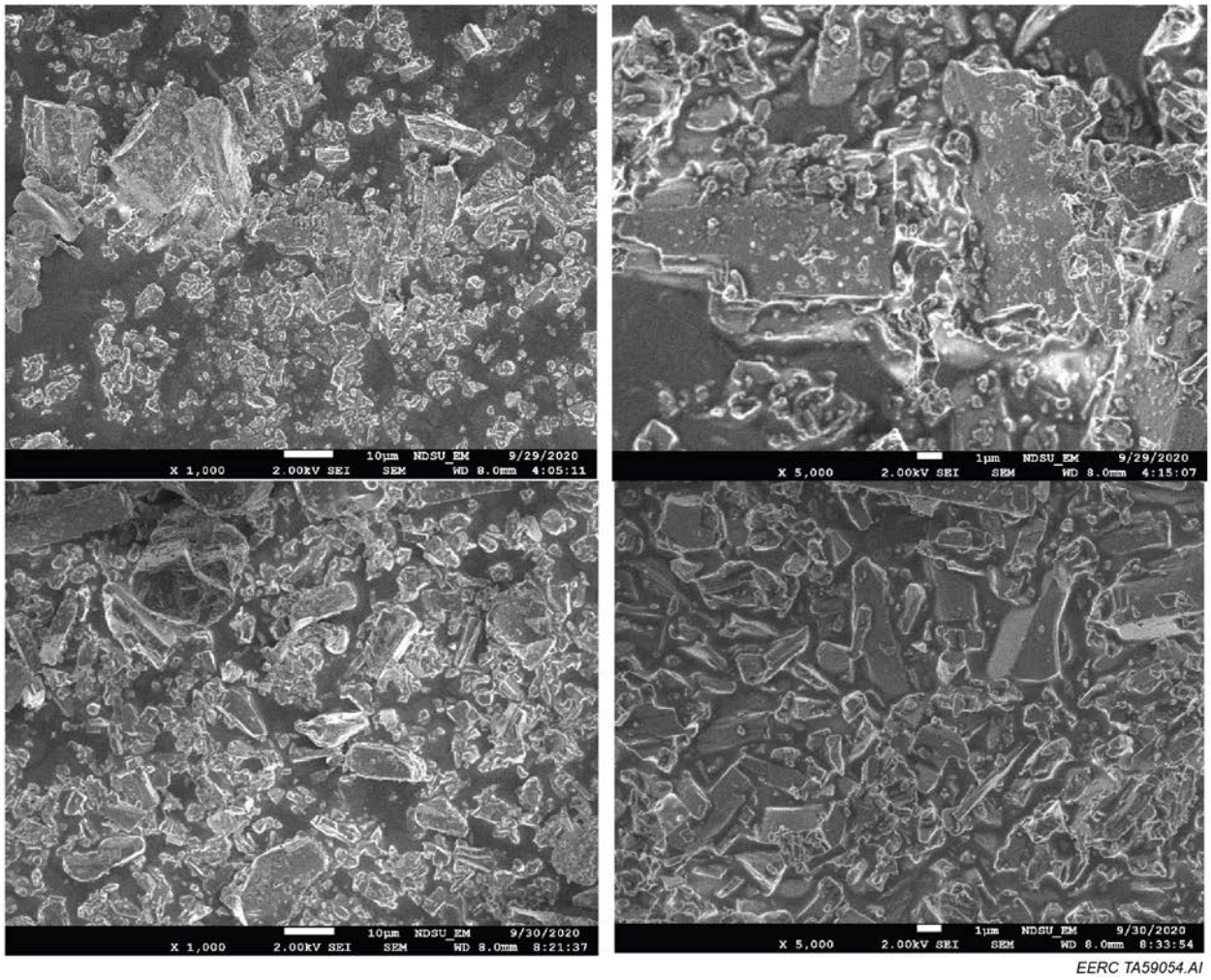

Figure 3. IPC2 particles before (top) and after (bottom) 20 minutes of probe sonication.

power of 500 watts and frequency of 20 kilohertz. The photos show that while large particles were reduced in size, further bulk particle size reduction—possibly achievable through an optimum combination of sonication time, power, and frequency—is needed. If additional probe sonication work yields SEM evidence indicating meaningful reduction of bulk IPC2 particle size, reduction magnitude will be quantified using a dynamic light-scattering (DLS) technique. Access to the DLS instrument is currently limited by COVID-19 risk mitigation protocols.

Work is progressing on optimizing a procedure for hot-pressing thin disks of IPC2 using PBI powder as binder. The objective is to eliminate voids/cavities and increase IPC2 particle packing density versus solution/film-cast membranes. In addition, it is anticipated that an optimized heating–cooling regimen would yield a high-density disk that retains residual compressive stresses upon cooling. Much like in prestressed concrete, these residual stresses should significantly increase disk strength and thereby allow manufacture of thinner disks with high integrity. The hot press system was utilized to monitor the impact of temperature on deformation of pure PBI. Figure 4 illustrates the impact of increasing temperature on compression of pure PBI under a constant pressure of 100 psi. The data indicate the onset of PBI densification as temperature reaches approximately 700°F, significantly below the PBI glass transition temperature of 800°F.

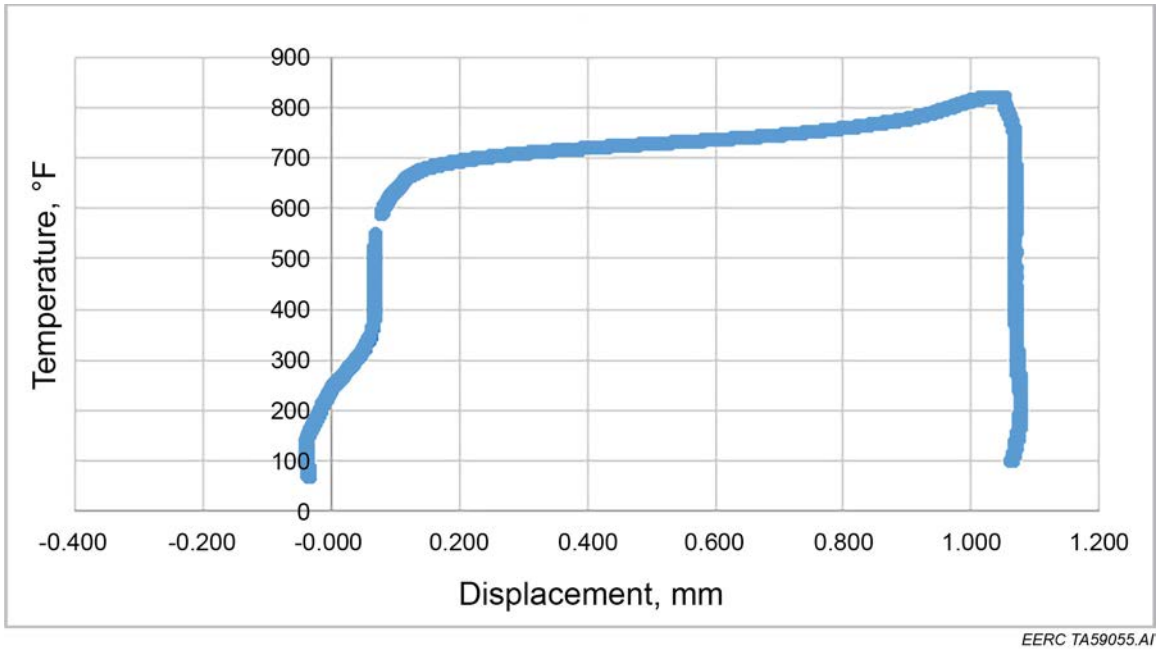

Figure 4. PBI powder compression versus temperature at 100 psi fixed stress.

As described in the last quarterly progress report, work is ongoing to identify an inorganic IPC2 binder that offers higher performance than PBI, which has zero proton conductivity. Two promising inorganic materials with reported glass transition temperatures well below that of IPC2 were synthesized. Based on their compositions, the binders have potential for significant proton conductivity. The binders are awaiting differential scanning calorimetry to definitively

establish their glass transition temperatures, after which thin disks will be prepared (via heat-pressing mixtures of IPC2 and each binder) and tested for proton conductivity at 300°C.

A method was developed to strategically modify IPC2 particle surfaces, with the objective of increasing IPC2 proton conductivity. The resulting material, referred to as IPC2-A2, was compared to IPC2 based on proton conductivity at 300°C. Tests were conducted using disks pressed from "doughs" comprising a viscous PBI-in-DMAc solution with added particles of either IPC2 or IPC2-A2. After pressing at 15,000 psi for 10 minutes, the disks were dried under ambient air for 30 hours. During conductivity testing, both disk types were exposed to humidified hydrogen on the anode side and humidified nitrogen on the cathode side, with all gases supplied at identical rates and absolute humidity levels. As shown in Table 2, IPC2-A2 exhibited about 57% more proton conductivity than IPC2. Also noteworthy was the time required for disk saturation. While IPC2 typically requires about 90 minutes for saturation, IPC2-A2 appeared to reach saturation in about 20 minutes. Quicker saturation may mean that less humidity is required to sustain high proton conductivity. Additional testing is needed to confirm IPC2-A2 conductivity and ensure its sustainability.

Table 2. IPC2 versus IPC2-A2 – Proton Conductivity Comparison

Disk Type	Fabrication Method	IPC/PBI Mass Ratio	Proton Conductivity (S/cm)
IPC2	Dough pressed	90/10	0.7E-2
IPC2-A2	Dough pressed	90/10	1.1E-2

Surface oxidation of the sample holder used for measuring membrane proton conductivity at 300°C was recently identified as the source of inaccurate conductivity data. An appropriate sample holder (referred to as "ProboStat™") designed and fabricated by NORECS (a University of Oslo spin-off company specializing in equipment for characterizing electrical properties of materials at high temperatures) is available for $24K. The EERC requested approval to purchase the ProboStat with available project funds. Following approval, delivery is expected to take at least 8 weeks.

Task 5 – Catalyst Screening and MEA/Unit Cell Development and Optimization

A set of MEAs comprising membrane (75% IPC2–25% PBI), niobium nitride cathode, and platinum anode were fabricated. Application of electrodes to membrane was performed using an airbrushing technique. Because airbrushing typically requires an ionomer dispersion similar in viscosity to water, significant experimentation was required to establish an appropriate mixture. To achieve the target viscosity, a relatively viscous mixture of PBI dissolved in DMAc was prepared, and viscosity was adjusted via incremental DMAc addition and specific gravity measurement with a pycnometer. An appropriate amount of IPC2 was then added, and the mixture was stirred with a magnetic stir bar in conjunction with probe sonication, required to achieve a well-disbursed suspension of IPC2 particles, since IPC2 is insoluble in DMAc. After more experimentation, an ink formula was established that included catalyst (platinum black or niobium nitride), the ionomer dispersion, and additional DMAc. After ink preparation and

vigorous mixing (to ensure IPC2 particle suspension), inks were airbrushed onto membrane surfaces using a waka airbrush with a nitrogen gas flow. Airbrushing was conducted (using a hot plate) at 200°C to evaporate DMAc while leaving the catalyst and ionomer dispersion on the membrane.

Key to successful airbrushing of electrodes onto membrane was establishing an efficient way to hold the membrane against the hot plate during the airbrushing process. Early trials proved that tape (used to hold membranes in place) could not be easily removed from the membrane without ripping it. Therefore, a holder was created out of carbon paper, which held the membrane against the hot plate, while restricting the surface exposed to tape to the carbon paper, as displayed in Figure 5, to avoid this issue. This type of holder also allows the shape and size of the electrode to be controlled.

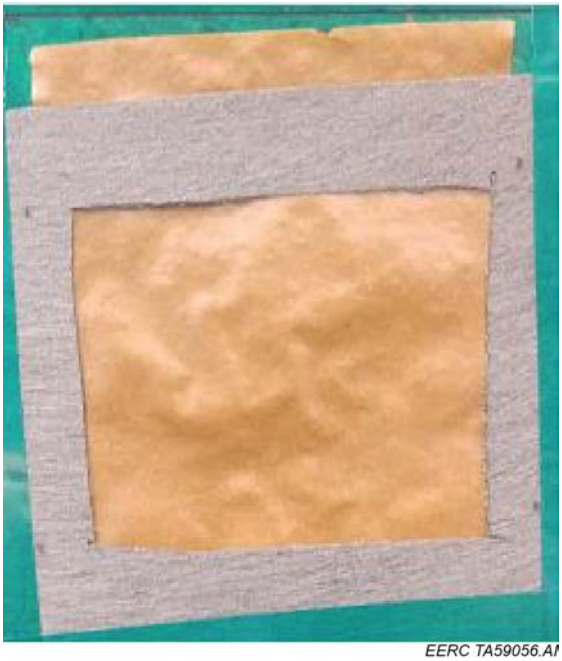

Figure 5. Carbon paper membrane holder for electrode application via airbrushing.

MEA test samples were created using the carbon paper holder and evaluated for electrode adhesion (to membrane) strength. Adhesion was evaluated by soaking MEAs and uncoated membranes in water for over an hour before patting the membranes dry with a paper towel. As the electrode remained on the membrane during and after soaking, it was determined that the electrode was well adhered. Although good adhesion was demonstrated, these first attempts at airbrush coating revealed that membrane texture (in the form of wrinkles) was a problem, since wrinkles resulted in pooling of electrode ink and uneven electrode thickness. To attempt removal of membrane texture, a series of heat-pressing trials was undertaken using a Carver press at differing temperature–pressure regimes. The testing established that a smooth membrane could be produced by pressing it between Teflon paper sheets and metal plates (top and bottom) at

150°C and 1000 pounds of pressure for 10 minutes, as shown in Figure 6. Smoothed membranes were used for subsequent fabrication of MEAs. To make a series of identical MEAs for evaluation based on ammonia synthesis rate and current efficiency, a specialized stencil/sample holder was created, as shown in Figure 7.

Figure 6. Film-cast membranes as produced (left), after pressing at 500 pounds and 65°C for 5 minutes (center), and 1000 pounds and 150°C for 10 minutes (right).

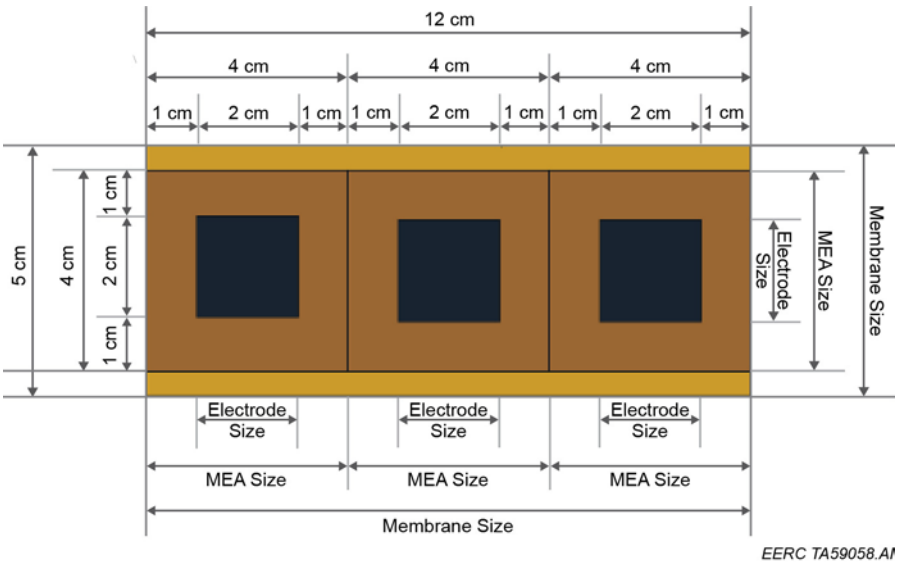

Figure 7. Schematic of stencil/membrane holder for producing multiple electrodes/MEAs from a single membrane sample.

Task 6 – Design, Fabrication, and Operation of 100-g/d LPEA System

No activity this quarter.

Task 7 – Techno-Economic Analysis

No activity this quarter.

PLANS FOR NEXT QUARTER

Task 3 – Optimize IPC and PIC Membrane Performance and Durability

Evaluation and—as warranted—optimization of probe sonication and planetary ball milling methods for IPC2 particle size reduction will continue.

Work will continue on development of methods for producing thin, densified, "pre-stressed" PIC electrolyte disks via hot-pressing mixtures of:

- IPC2 and PBI binder
- IPC2 and ceramic/inorganic binders
- IPC2-A2 and PBI binder
- IPC2-A2 and ceramic/inorganic binders

Efforts to procure and/or develop a membrane sample holder unsusceptible to surface oxidation and other electrical conduction/resistance issues that impede accurate measurement of high-temperature proton conductivity will continue. In addition to pursuing procurement of the ProboStat sample holder for membrane proton conductivity measurement at 300°C, the EERC will explore the possibility of utilizing a recently acquired Fiaxell solid oxide fuel cell (SOFC) test system for this purpose. Although designed for evaluation of small active-area SOFC electrolytes and cells, based on initial assessment and discussions with Fiaxell technical staff, it appears likely that the test stand could be deployed for accurate measurement of proton exchange membrane conductivity.

Task 5 – Catalyst Screening and MEA/Unit Cell Development and Optimization

Additional MEAs (including MEAs with different cathode catalysts) will be fabricated and evaluated based on ammonia synthesis rate and current efficiency.

Task 6 – Design, Fabrication, and Operation of 100-g/d LPEA System

Initiate design of 100-g/d system.

Task 7 – Techno-Economic Analysis

Develop strategy/deployment scenario for economically competitive initial entry of LPEA into the commercial ammonia industry.

PRODUCTS

None.

IMPACTS

Impact on Technology Transfer and Commercialization Status

No commercialization impacts, progress, issues, or concerns to report during this quarter.

Dollar Amount of Award Budget Being Spent in Foreign Country(ies)

No spending of any project funds in any foreign countries has occurred or is planned.

CHANGES/PROBLEMS

The EERC is operational and open for business. Personnel that are not essential for on-site operations have transitioned to working from home. Essential project, laboratory, and field-based activities are proceeding with the incorporation of the Centers for Disease Control and Prevention, North Dakota State, and UND guidelines associated with COVID-19, and mitigation measures have been implemented.

In collaboration with project partners, the EERC is continually assessing potential impacts to project activities resulting from COVID-19 and/or the U.S. economic situation.

As discussed earlier and shown in Table 3 (Milestone Schedule), the project is about 6 months behind schedule.

Scope Issues, Risks and Mitigation Strategies

None.

Actual or Anticipated Problems or Delays and Corrective Actions or Plans to Resolve Them

The project is behind schedule due in large part to laboratory staffing restrictions imposed to minimize COVID-19 spread. Although restrictions were eased for several months, an ongoing surge in North Dakota cases has resulted in their reimplementation until further notice.

Changes That Have a Significant Impact on Expenditures

None.

RECIPIENT AND PRINCIPAL INVESTIGATOR DISCLOSURES

None.

CONFLICTS OF INTEREST WITHIN PROJECT TEAM

None.

PARTNERS AND FINANCIAL INFORMATION

This project is sponsored by NDIC, DOE, UND Chemistry, NDSU, and Proton. Table 3 shows the total budget of $3,164,010 for this project and expenses through the reporting period.

Table 3. Project-to-Date Financial Report at September 30, 2020

Funding Source	Budget	Current Reporting Period Expenses	Cumulative Expenses as of 9/30/20	Remaining Balance
DOE	$2,497,983	$161,520	$1,680,806	$817,177
UND Chemistry – In Kind	$69,027	$6,985	$68,265	$762
NDIC	$437,000	$24,137	$315,984	$121,016
NDSU – In Kind	$120,000	$0	$120,000	$0
Proton – In Kind	$40,000	$1,895	$15,402	$24,598
Total	**$3,164,010**	**$194,537**	**$2,200,457**	**$963,553**

REFERENCE

Columban, P. (Editor), Proton Conductors: Solids, Membranes and Gels – Materials and Devices (Volume 2 of Chemistry of Solid-State Materials) 1st Edition, 1992.